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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

9052-98

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

09/980219

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

INTERNATIONAL APPLICATION NO.
PCT/GB00/02059

INTERNATIONAL FILING DATE
26 May 2000

PRIORITY DATE CLAIMED
2 June 1999

TITLE OF INVENTION

ADHESIVE COMPOSITION COMPRISING THERMOEXPANDABLE MICROCAPSULES

APPLICANT(S) FOR DO/EO/US
Peter Stewart Bain, Giovanni Manfre

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☐ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☒ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
- ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☐ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4)
- ☒ Amendments to the claims of the International Application Under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
- ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
- ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- ☐ An English language translation of the annexes of the International Preliminary Examination Report Under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☒ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4)
20. ☐ Other items or information:

U.S. APPLICATION NO. 09/980219		INTERNATIONAL APPLICATION NO. PCT/BG60/02059		ATTORNEY DOCKET NO. 9052-98	
21. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492(a)(1) - (5)):				CALCULATIONS PTO USE ONLY	
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO.....				\$1040.00	
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO.....				\$890.00	
International preliminary examination fee 37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....				\$740.00	
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4).....				\$710.00	
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4).....				\$100.00	
ENTER APPROPRIATE BASE FEE AMOUNT =				\$890.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	32 - 20 =	12	x \$18.00	\$216.00	
Independent Claims	8 - 3 =	5	x \$84.00	\$420.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$280.00	\$	
TOTAL OF ABOVE CALCULATIONS =				\$1526.00	
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2				\$	
SUBTOTAL =				\$1526.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$1526.00	
Fee for Recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +				\$	
TOTAL FEES ENCLOSED =				\$1526.00	
				Amount to be refunded:	\$
				charged:	\$

- a. ☒ A check in the amount of \$1,526.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 50-0220 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-0220. A duplicate copy of this sheet is enclosed.
- d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:



20792

PATENT TRADEMARK OFFICE

Jarett K. Abramson
Jarett K. Abramson, #47,376
Date: Nov. 29, 2001

CERTIFICATE OF EXPRESS MAILING

Express Mail Label No. EL 920742868 US

Date of Deposit: November 29, 2001

I hereby certify that this correspondence is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to: BOX PCT, U.S. Patent and Trademark Office, P.O. Box 2327, Arlington, VA 22202, Attn: DO/EO/US.

Susan E. Freedman
Susan E. Freedman

Attorney's Docket No. 9052-98

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re:	Bain et al.	Examiner:	To be assigned
Serial No.:	To be assigned	Group Art Unit:	To be assigned
Filed:	Concurrently		
For:	ADHESIVE COMPOSITION COMPRISING THERMOEXPANDABLE MICROCAPSULES		

Date: November 29, 2001

Box PCT
U.S. Patent and Trademark Office
P.O. Box 2327
Arlington, VA 22202
Attn: DO/EO/US

PRELIMINARY AMENDMENT

Sir:

Prior to the examination of the above application, please amend the above referenced application as follows. Please enter the following amendments prior to the calculation of the filing fee.

IN THE SPECIFICATION:

Please amend the specification as follows:

On page 1 after the title of the invention please add:

RELATED APPLICATIONS

This application claims priority under 35 U.S.C. § 371 from PCT/GB00/02059, (published under PCT Article 21(2) in English), filed on May 26, 2000, which claims priority to Great Britain Application Serial No. 9912694.8, filed on June 2, 1999, the disclosures of which are incorporated by reference herein in their entireties.

On page 1 please replace the second full paragraph with the following:

Typically to install a window pane in a wooden/plastic/metal frame, the glass pane is firstly held in position against nails or other clasps and then fixed into position by putty or plasters material. Conventional putty is a cement made from whiting and

linseed oil which hardens over time to provide a peripheral rim of the window pane, thus separating interior and exterior environments and preventing air, moisture and/or heat transfer. The installation is completed once the putty has dried and this usually takes up to 6 hours or so depending on the kind of plasters used.

IN THE CLAIMS:

Please amend the claims as follows:

2. (Amended) A composition according to Claim 1 wherein the microcapsules each comprise a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

4. (Amended) A composition according to Claim 1, wherein the adhesive agent is selected from the group consisting of urethane, polyurethane, polyvinylchloride and an MS polymer.

5. (Amended) A composition according to Claim 1, wherein the thermoexpandable capsules are microspheres or hollow fibres in the form of a powder.

7. (Amended) A composition according to Claim 1, wherein the composition is formed at the time of, or shortly before, its use.

9. (Amended) A composition according to Claim 1, which further includes a fast cure agent or catalyst, whereby the composition is rapidly cured or set.

10. (Amended) A composition according to Claim 1, which further includes a coloring agent so that the cured composition is black.

11. (Amended) A composition according to Claim 1, wherein the microcapsules encapsulate more than one material.

13. (Amended) A composition according to Claim 11 wherein the microcapsules encapsulate different agents, either separately or in combination.

14. (Amended) A composition according to Claim 11, further comprising intact expanded microspheres and/or microcapsules which have released their contents into the composition.

15. (Amended) A composition according to Claim 2, wherein the microcapsule shell diameter is in the range 10 to 120 μm .

16. (Amended) A composition according to Claim 2, wherein the microcapsule shell thickness is in the range 3 to 7 μm .

17. (Amended) A composition according to Claim 1, wherein the thermoexpandable microcapsules are present in the range of 1-30% by volume

19 (Amended) A composition according to Claim 1 wherein the composition is activated by heat in a heat activation range of 80-170° C.

21. (Amended) A composition according to Claim 2, further comprising a mixture of microcapsules of different diameter or shell thickness or of differing heat activation temperatures or different expanding coefficients.

22. (Amended) A method of preparing a composition comprising:
providing an adhesive agent; and
dispersing thermoexpandable microcapsules therein for fixing glazing.

23. (Amended) The method according to Claim 22 wherein the composition further comprises a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

25. (Amended) A composition according to Claim 24 further comprising a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

26. (Amended) A method of preparing a composition comprising:
providing a primer; and
dispersing thermoexpandable microcapsules therein as a glazing adhesive.

27. (Amended) The method according to Claim 26 further comprising a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

28. (Amended) A method of preparing a glazing adhesive comprising:
providing a composition comprising microcapsules.

29. (Amended) A method of installing a vehicle windscreen comprising:
(i) placing a windscreen flush against a window aperture rim of a vehicle;
(ii) applying a composition comprising an adhesive agent and dispersed therein thermoexpandable microcapsules around a peripheral area of the windscreen; and
(iii) allowing sufficient time for the adhesive to cure or primer to dry.

31. (Amended) A method of fixed glazing comprising:
(i) applying a heat source to the cured adhesive wherein the heat applied is sufficient to cause thermoexpansion of the microcapsules and to weaken the adhesive cohesion and interface bonds of the composition; and
(ii) removing the windscreen from the main vehicle body.

32. (New) A method of installing a vehicle windshield comprising:
placing a windshield flush against a window aperture rim of a vehicle,
applying a composition comprising a primer and dispersed therein
thermoexpandable microcapsules around a peripheral area of the windshield;
and
allowing sufficient time for the adhesive to cure or primer to dry.

REMARKS

Please note that the claims pending at the time of this filing are the claims of the international application serial no. PCT/GB00/02059, *i.e.* claims 1-31. The pending claims have been amended above to better conform to United States practice. The marked-up version of the changes to the specification and claims are attached hereto in the "Version With Markings to Show Changes Made".

It is submitted that this application is now in condition for substantive examination, which action is respectfully requested.

Respectfully submitted,



Jarett K. Abramson
Attorney for Applicants
Registration No. 47,376

Enc: Version With Markings to Show Changes Made

CUSTOMER NO.



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PATENT TRADEMARK OFFICE

CERTIFICATE OF EXPRESS MAILING

Express Mail Label No. EL 920742868 US

Date of Deposit: November 29, 2001

I hereby certify that this correspondence is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to: BOX PCT, U.S. Patent and Trademark Office, P.O. Box 2327, Arlington, VA 22202, Attn: DO/EO/US.



Susan E. Freedman

Date of Signature: November 29, 2001

VERSION WITH MARKINGS TO SHOW CHANGES MADE

IN THE SPECIFICATION:

On page 1 after the title of the invention please add:

RELATED APPLICATIONS

This application claims priority under 35 U.S.C. § 371 from PCT/GB00/02059, (published under PCT Article 21(2) in English), filed on May 26, 2000, which claims priority to Great Britain Application Serial No. 9912694.8, filed on June 2, 1999, the disclosures of which are incorporated by reference herein in their entireties.

On page 1 please replace the second full paragraph with the following:

Typically to install a window pane in a wooden/plastic/metal frame, the glass pane is firstly held in position against nails or other clasps [and] and then fixed into position by putty or plasters material. Conventional putty is a cement made from whiting and linseed oil which hardens over time to provide a peripheral rim of the window pane, thus separating interior and exterior environments and preventing air, moisture and/or heat transfer. The installation is completed once the putty has dried and this usually takes up to 6 hours or so depending on the kind of plasters used.

IN THE CLAIMS:

A marked up version of each of the presently amended claims, highlighting the changes thereto, follows:

2. (Amended) A composition according to Claim 1 wherein the microcapsules each [comprising] comprise a shell[, encapsulate] that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

4. (Amended) A composition according to [any preceding claim] Claim 1, wherein the adhesive agent is [a] selected from the group consisting of urethane, [or] polyurethane, [or] polyvinylchloride [or a] and an MS polymer.

5. (Amended) A composition according to [any preceding claim] Claim 1, wherein the thermoexpandable capsules are microspheres or hollow fibres in the form of a powder.

7. (Amended) A composition according to [any of Claims 1-5] Claim 1, wherein the composition is formed at the time of, or shortly before, its use.

9. (Amended) A composition according to [any preceding claim] Claim 1, which further includes a fast cure agent or catalyst, whereby the [adhesive] composition is rapidly cured or set.

10. (Amended) A composition according to [any preceding claim] Claim 1, which further includes a [colouring] coloring agent so that the cured composition is black.

11. (Amended) A composition according to [any preceding claim] Claim 1, wherein the microcapsules encapsulate more than one material.

13. (Amended) A composition according to [either] Claim 11 [or 12] wherein the microcapsules encapsulate different agents, either separately or in combination.

14. (Amended) A composition according to [any of claims 11-13] Claim 11, further comprising intact expanded microspheres and/or microcapsules which have released their contents into the composition.

15. (Amended) A composition according to [any preceding claim] Claim 2, wherein the [microsphere's] microcapsule shell diameter is in the range 10 to 120 μm .

16. (Amended) A composition according to [any preceding claim] Claim 2, wherein the microcapsule shell thickness is in the range 3 to 7 μm .

17. (Amended) A composition according to [any preceding claim] Claim 1, wherein the thermoexpandable microcapsules are present in the range of 1-30% by volume

19. (Amended) A composition according to [any preceding claim] Claim 1 wherein the composition is activated by heat in a heat activation range of 80-170° C.

21. (Amended) A composition according to [any preceding claim] Claim 2, further comprising a mixture of microcapsules of different diameter or shell thickness or of differing heat activation temperatures or different expanding coefficients.

22. (Amended) [Use of] A method of preparing a composition comprising;
providing an adhesive agent; and
[dispersed therein] dispersing thermoexpandable microcapsules therein for fixing glazing.

23. (Amended) [Use of a composition] The method according to Claim 22 wherein the composition further [including any one or more of the features recited in Claims 2-21] comprises a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

25. (Amended) A composition according to Claim 24 further [including any one or more of the features of Claims 2-21] comprising a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

26. (Amended) [Use of] A method of preparing a composition comprising;
providing a primer; and
[dispersed therein] dispersing thermoexpandable microcapsules therein as a glazing adhesive.

27. (Amended) [Use of a composition] The method according to Claim 26 [including any one or more of the features of Claims 2-21] further comprising a shell that encapsulates at least one expandable gas or volatile expandable agent or an explosive material.

28. (Amended) [A composition comprising microcapsules for use as a] A method of preparing a glazing adhesive comprising:
providing a composition comprising microcapsules.

29. (Amended) A method of installing a vehicle windscreen [or fixed glazing] comprising [the steps of]:

- (i) placing a windscreen flush against a window aperture rim of a vehicle;
- (ii) applying [the] a composition [according to any of Claims 1-21 or Claims 24, 25 or 28] comprising an adhesive agent and dispersed therein thermoexpandable microcapsules around a peripheral area of the windscreen;
and
- (iii) allowing sufficient time for the adhesive to cure or primer to dry.

31. (Amended) A method of [removing a vehicle windscreen or] fixed glazing comprising [the steps of]:

- (i) applying a heat source to the cured adhesive wherein the heat applied is sufficient to cause thermoexpansion of the microcapsules and [thus] to weaken the adhesive cohesion and interface bonds of the composition; and
- (ii) removing the windscreen from the main vehicle body.

32. (New) A method of installing a vehicle windscreen or fixed glazing comprising:

- placing a windscreen flush against a window aperture rim of a vehicle;
- applying a composition comprising a primer and dispersed therein thermoexpandable microcapsules around a peripheral area of the windscreen;
and
- allowing sufficient time for the adhesive to cure or primer to dry.

WO 00/75254

PCT/GB00/02059

ADHESIVE COMPOSITION COMPRISING THERMOEXPANDABLE MICROCAPSULES

The present invention relates to an adhesive for use in sealing together two surfaces, for use especially in the glazing industry in general and in securing vehicle
5 windscreens and/or windows and/or other vehicle features that may require replacement such as, without limitation, car panels; the invention also provides a method of use for installing and/or replacing vehicle windscreens and/or other fixed glazing on vehicles, buildings or the like.

10 Background of the Invention

Typically to install a window pane in a wooden/plastic/metal frame, the glass pane is firstly held in position against nails or other clasps and then fixed into position by putty or plasters material. Conventional putty is a cement made from whiting and
15 linseed oil which hardens over time to provide a peripheral rim of the window pane, thus separating interior and exterior environments and preventing air, moisture and/or heat transfer. The installation is completed once the putty has dried and this usually takes up to 6 hours or so depending on the kind of plasters used.

20 To remove a window pane after it has been fixed in position in a frame requires the window itself to be shattered so that the hardened putty or plasters can be scraped/chiselled away from the frame. The removal operation can cause damage to the frame and varnishes.

25 In use, the window pane is held rigidly around its edges so that even relatively small vibrational mechanical movements such as with earthquakes or bomb blasts or strong winds can cause the window pane to shatter.

In the automotive industry, cars direct from the factory production line typically have
30 the windscreens and other fixed windows, including light assemblies fixed into position by placing the glass against a frame rim and using adhesives so as to direct glaze the glass. The life span of a windscreen and other fixed windows are

significantly shorter than that of the vehicle itself partially due to degradation or damage or being deliberately broken by vandals/car thieves. Thus a motorist may need to replace the windscreen several times during the vehicle's lifetime.

- 5 Additionally, glued glazings have to be replaced any time the window, especially the windscreen, has been damaged in its optical performance by, for example, impact stones or other fractures or abrasion by wipers. Damage to the window surface can increase the scattering of light and may reduce the visibility to levels below safety limits. Moreover, regulations of motor worthiness (MOT) stipulate that there can be
- 10 no chips or visual impairments on laminated windscreens, so whereas recent improvements have made the windscreens shatter-proof, they are still prone to chipping and fracturing and thus will require replacement.

- The process of replacing vehicle windscreens is both laborious and time consuming.
- 15 The automotive glass fitter has first to remove the defective windscreen (usually in intact form), however the windscreen is firmly bonded in place and the adhesive sealant is hardened. Typically the fitter uses a device comprising a cheesewire. The cheesewire is used to cut/saw through the hardened rubber along the periphery of the windscreen. This process requires physical force and can lead to musculo-skeletal
- 20 conditions in the fitters themselves as a result of repetitive strain injury. Further problems associated with this method are that the cheesewires can overheat due to friction, additionally the wires themselves can break.

- Other methods of detaching the windscreen from the adhesive sealant include: the
- 25 use of mechanical oscillator knives/cutters to cut through the hardened material or; directed heat such as a laser beam to soften the sealant prior to removing the windscreen with either cheesewire or specialised bladed tools. The problem with a method where heat is directly applied to the sealant is that the heat required to soften the hardened adhesive sealant can concomitantly and inadvertently damage the
- 30 vehicle's paintwork and/or other exterior surfaces. For example, a pulsed laser that is

set to pulse too fast will not generate enough energy to char the adhesive sealant and a pulsed laser that is set too slow will burn the adhesive sealant and liquify it.

Once the windscreen has been freed from the rubber sealant it can be removed and the surround scraped before it is replaced. It is known from the prior art to use urethane based adhesives to fix/seal the replaced windscreen in place and to apply the adhesive from a dispenser gun to specific peripheral edges so as not to impinge on the viewing capacity of the windscreen. The adhesive typically takes about 8 hours to cure.

Recent advances to the industry have provided for the inclusion of fast cure agents/catalysts so as to speed up the time from vehicle drop-off to vehicle collection. The fast cure agents/catalysts can be provided pre-mixed in the adhesive composition or alternatively can be mixed with the adhesive at the point of exit from a dispensing gun. However the problem still remains that the removal of a defective windscreen and its subsequent replacement is a laborious and time consuming process which can result in damage to the dashboard interior or vehicle paint-work.

An adhesive that could satisfy vehicle safety crush and crash standards and provide for easy, effective and damage-proof removal of a defective windscreen or other fixed glazing from a vehicle would offer immediate improvement to the industry and consumer.

In a completely different technical field it is known to provide thermoexpandable microcapsules or microspheres for use in the manufacture of porous or lightweight materials with density (weight) reduction, acoustic and thermal insulating properties as covering materials or walls. The microcapsules or microcapsules comprises a polymer shell or shell of some other similar material, the shell being of certain thickness and chemical/physical/mechanical properties. The shell encapsulates materials such as volatile organic solvents, expandable gases or activating agents, including explosives or any other such material which is capable of exploding the

shell and expanding at certain specified conditions with a selected matrix . The microcapsules, when heated to a sufficient temperature, typically in the region of about 75-180 ° C, depending on the encapsulated substance and the shell composition and thickness, can produce an increase of their volume at high expansion rate in some instances at a volumetric expansion limit of up to 70 to 160 times the original volume. The percentage and distribution of the microspheres in a given composition, their expansion ratio, the temperature operating range, the softening transition range of the shell material and the matrix cohesion and consistency are all parameters which are able to influence the expansion volume of a layer between two materials.

10 We have discovered that by mixing an appropriate adhesive in a suitably rheological performance with a certain % in powdered form of specially developed microcapsules having a specific range of size distribution, the resulting composition is effective at providing glazing adhesion and sealing. Moreover, of particular
15 advantage is that the adhesive bonds in the composition can be weakened by the application of direct heat to the composition thereby allowing sufficient softening of the adhesive material so that a vehicle windscreen/fixed glazing can be easily lifted up and so rapidly removed. The lifting pressure can be predicted by a computational software which is part of this invention and treats the expanding microspheres like a
20 spring mechanical pressure actuator in the volume of the adhesive state and like a "bombing" actuator at interfaces between two layers.

We believe that the invention provides the first application/use of a polyester material in the automotive glazing industry.

25 It will be appreciated that the adhesive of the invention has application in other areas especially where two surfaces are to be bonded together and where one surface may subsequently need replacing following damage or wear, for example, and without limitation; shower doors and vehicle panels and other building glazing applications.

30

Reference herein to vehicle is intended to include, without limitation car, lorry, van ship, boat, plane, cable car, helicopter, hovercraft and any other form of transport in which there is fixed glazing.

5 Statement of the Invention

In its broadest aspect the invention provides a composition comprising a heat triggered adhesion-deactivating microcapsule or microsphere or microbead, typically the microcapsule or microsphere or microbead is typically dispersed in another
10 medium such as an adhesive or primer or at an interface between two layers.

According to a first aspect of the invention there is provided a composition comprising an adhesive agent and dispersed therein thermoexpandable microcapsules.
15

Reference herein to microcapsule is intended to include a microsphere or microbead.

Preferably, the microcapsules each comprise a shell which encapsulates at least one expandable gas or volatile expandable agent or an explosive material. Preferably, the
20 shell is composed of a polymer or other suitable material.

Preferably, the adhesive agent is a urethane or polyurethane or polyvinylchloride or a MS polymer.

25 Preferably, the thermoexpandable capsules are microspheres or hollow fibres in the form of a powder of specific range of dimensions and properties.

The powder can be provided with the adhesive agent in a pre-mixed form in a container or the powder can be introduced into and mixed with the adhesive or one
30 component of it at a point of exit from a dispensing device i.e. the composition can be formed as a pre-mix or at the time of, or shortly before, its use. The dispensing

device can be followed by a static mixer suitably lubricated at the point of exit of the composition for optimal blending.

Preferably, the composition additionally comprises a fast cure agent or catalyst,
5 whereby the adhesive composition is rapidly cured or set. Preferably, the composition comprises a colouring agent so that the cured composition is black.

Reference herein to cure is intended to mean the hardening or setting of the adhesive mixture, the hardening or setting can be either chemically or non-chemically
10 enhanced.

Preferably, the microcapsules encapsulate more than one material, ideally the material is selected from the group consisting of an expanding agent, an agent capable of sublimation, water, an explosive material or an activator agent.
15

Preferably, the activator agents are capable of foaming or of shrinkage. The present invention includes the simultaneous use of microcapsules encapsulating a variety of different agents, either separately or in combination. The additional microcapsules are activated by the breaking or permeation of the polymer shell whereby their
20 contents are released so as to interact with the adhesive mix. Their activation is as a result of specified applied conditions and thus is controllable. Microspheres do not break/fracture their shells in the expanding state maintaining their integrity, so that an activated composition comprises intact expanded microspheres and microcapsules which have released their contents into the matrix composition.
25

It will be appreciated that the expanding agent inside the capsule is capable of activating a foaming process of the adhesive composition and that the agent capable of sublimation is to allow the composition to expand under certain specified conditions. Both these processes will occur after the breakage/fracture of the
30 microcapsule shell and thus contribute to facilitating lifting and ease of removing fixed glazing. The inclusion of water in the microcapsules is to allow the adhesive

composition to weaken in certain conditions and the presence of an activator agent is to crosslink or polymerise the adhesive composition whereby shrinkage occurs and the adhesive composition weakens.

- 5 Preferably, the microsphere's diameter is in the range 10 to 120 μm .

Preferably, the microcapsule shell thickness is in the range 3 to 7 μm .

- 10 Preferably, the composition comprises microcapsules in the range of 1-30% by volume, and more preferably in the range of 2-10% by volume.

Preferably, the composition is activated by heat wherein the heat activation range is 80-170° C and ideally 120-150° C.

- 15 Preferably, the composition is composed of a mixture of microcapsules of different diameter or shell thickness or of differing heat activation temperatures or different suitable expanding coefficients.

- 20 The temperature of a vehicle windscreen can reach over 100°C in natural soak conditions. Thus the heat activation of the microcapsules in the composition of the invention needs to be in excess of any natural temperatures that may occur whilst being below those that could damage a vehicle dashboard trim or paint-work. It is envisaged that the composition of the invention will have application in many fields and many diverse climates hence the composition may be provided with selected heat
- 25 activation ranges depending on its intended use and/or country of use for both kinds of microcapsule: the expanding microspheres and the microcapsules with breakable shells. For example, in the instance of the composition being used for fixing and sealing a shower door it is envisaged that the adhesive composition microcapsule heat activation range would be in the region of 80-100 °C, whilst for the majority of
- 30 automotive glazing the microcapsule heat activation range would be in the region of

120-150° C. Optionally in either composition a user may require microcapsules capable of releasing a curing agent and/or an activator agent capable of shrinkage.

Heat activation of the microcapsules causes the beads/fibres to thermoexpand thus creating pressure along the rim of glazing. This in effect reduces the viscosity and the shear or tear stress of the adhesive material. In addition, heat activation or expansion of the microcapsules reduces the cohesive stress and/or interfacial adhesive bonding of the adhesive film between two bonded surfaces, typically the frame and glass layers. The reduction in either cohesive stress and interfacial adhesion, or both together, contribute to reduce failure stress so as to facilitate removal of the glued glazing from the frame once the microcapsules have expanded at the specific temperature range and range of time, typically no more than 5 minutes. The compositions of the present invention thus allows removal of glued glazing by virtue of a reduction in chemical and/or physical bonding of the adhesion at the interface between the adhesive and the two bonded surfaces (glass and frame surfaces). In other words, adhesion failure can only occur at the interface of the two bonded surfaces due to the effect of the expanded microcapsules. In use, and once the microcapsules have been activated the reduction in cohesive forces of the adhesive material and the bonding at interface between material and layers in addition to the expansion of the adhesive material itself, results in the loosening of the whole of the adhesion of the glazing to a frame so that a windscreen or glazing can be easily lifted up and removed therefrom.

According to a further aspect of the invention there is provided use of a composition comprising an adhesive agent and dispersed therein thermoexpandable microcapsules as a glazing adhesive.

Preferably, the composition further includes any one or more of the features herein before described.

According to a yet further aspect of the invention there is provided a composition comprising a primer and dispersed therein thermoexpandable microcapsules.

Preferably, the composition further includes any one or more of the features herein before described.

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According to a further aspect of the invention there is provided use of a composition comprising a primer and dispersed therein thermoexpandable microcapsules as a glazing adhesive.

10 Preferably, the composition further includes any one or more of the features herein before described.

It will be appreciated that the invention also includes the use of microcapsules when
15 applied directly to a glazing face and/or body of a vehicle.

According to a yet further aspect of the invention there is provided a method of installing and/or replacing a vehicle windscreen or fixed glazing comprising the steps of:

- 20 (i) placing a windscreen flush against a window aperture rim of a vehicle,
(ii) applying the composition of the first aspect of the invention around a peripheral region of the windscreen;
(iii) allowing sufficient time for the adhesive to cure;
(iv) applying a heat source to the cured adhesive wherein the heat applied
25 is sufficient to cause thermoexpansion of the microcapsules and thus weaken the adhesive cohesion and interface bonds of the composition;
and
(v) removing the windscreen from the main vehicle body.

30 It will be appreciated that in one aspect, the method of installation involves steps i-iii whilst in another aspect the method of replacement involves steps iv-v.

By using the method of the invention as herein described, a vehicle windscreen or fixed glazing can be removed and/or replaced more rapidly and advantageously with less damage to the frame and/or paint work than by prior art methods, thus the method is more cost effective to both the fitter and customer.

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Claims

1. A composition comprising an adhesive agent and dispersed therein adhesion-deactivating thermoexpandable microcapsules.
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2. A composition according to Claim 1 wherein the microcapsules each comprising a shell, encapsulate at least one expandable gas or volatile expandable agent or an explosive material.
- 10
3. A composition according to Claim 2 wherein the shell is composed of a polymer.
4. A composition according to any preceding claim wherein the adhesive agent is a urethane or polyurethane or polyvinylchloride or a MS polymer.
- 15
5. A composition according to any preceding claim wherein the thermoexpandable capsules are microspheres or hollow fibres in the form of a powder.
- 20
6. A composition according to Claim 5 wherein the powder is provided with the adhesive agent in a pre-mixed form in a container.
7. A composition according to any of Claims 1-5 wherein the composition is formed at the time of, or shortly before, its use.
- 25
8. A composition according to Claim 7 wherein formation of the composition occurs within a dispensing device or at a point of exit therefrom.
9. A composition according to any preceding claim which further includes a fast
- 30
- cure agent or catalyst, whereby the adhesive composition is rapidly cured or set.

10. A composition according to any preceding claim which further includes a colouring agent so that the cured composition is black.
11. A composition according to any preceding claim wherein the microcapsules
5 encapsulate more than one material.
12. A composition according to Claim 11 wherein the additional material is selected from one or more of the group consisting of an expanding agent, an agent capable of sublimation, water, an explosive material or an activator agent.
- 10 13. A composition according to either Claim 11 or 12 wherein the microcapsules encapsulate different agents, either separately or in combination.
14. A composition according to any of claims 11-13 comprising intact expanded
15 microspheres and/or microcapsules which have released their contents into the composition.
15. A composition according to any preceding claim wherein the microsphere's diameter is in the range 10 to 120 μm .
- 20 16. A composition according to any preceding claim wherein the microcapsule shell thickness is in the range 3 to 7 μm .
17. A composition according to any preceding claim wherein the microcapsules
25 are present in the range of 1-30% by volume.
18. A composition according to Claim 17 wherein the microcapsules are present in the range of 2-10% by volume.
- 30 19. A composition according to any preceding claim wherein the composition is activated by heat in a heat activation range of 80-170° C.

20. A composition according to Claim 19 wherein the composition is activated by heat in a heat activation range of 120-150° C.
21. A composition according to any preceding claim comprising a mixture of microcapsules of different diameter or shell thickness or of differing heat activation temperatures or different expanding coefficients.
22. Use of a composition comprising an adhesive agent and dispersed therein adhesion-deactivating thermoexpandable microcapsules for fixing glazing.
23. Use of a composition according to Claim 22 wherein the composition further including any one or more of the features recited in Claims 2-21.
24. A composition comprising a primer and dispersed therein adhesion-deactivating thermoexpandable microcapsules.
25. A composition according to Claim 24 further including any one or more of the features of Claims 2-21.
26. Use of a composition comprising a primer and dispersed therein adhesion-deactivating thermoexpandable microcapsules as a glazing adhesive.
27. Use of a composition according to Claim 26 including any one or more of the features of Claims 2-21.
28. A composition comprising adhesion-deactivating microcapsules for use as a glazing adhesive.
29. A method of installing a vehicle windscreen or fixed glazing comprising the steps of:
- (i) placing a windscreen flush against a window aperture rim of a vehicle;

- (ii) applying the composition according to any of Claims 1-21 or Claims 24, 25 or 28 around a peripheral area of the windscreen; and
- (iii) allowing sufficient time for the adhesive to cure or primer to dry.

5 30. A method of removing a vehicle windscreen or fixed glazing comprising the steps of:

- (i) applying a heat source to the composition according to any one of claims 1 to 21, 24, 25 or 28 wherein the heat applied is sufficient to cause thermoexpansion of the microcapsules and thus weaken the adhesive cohesion and interface bonds of the composition; and
- (ii) removing the windscreen from the main vehicle body.

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(54) Title: ADHESIVE COMPOSITION COMPRISING THERMOEXPANDABLE MICROCAPSULES

(57) Abstract: The invention relates to a composition, its use and a method of its use as a glazing adhesive. The composition comprises an adhesive agent with thermoexpandable microcapsules which act as pressure actuators dispersed therein. The microcapsules are heat triggered so as to release at least one expandable volatile agent encapsulated within the microcapsule shell.

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DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

Attorney Docket No. 9052-98

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **ADHESIVE**,

the specification of which

☐ is attached hereto

OR

☒ was filed on May 26, 2000 as United States Application No. or PCT International Application Number PCT/GB00/02059 and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, §1.56, including material information that became available between the filing date of the prior application and the National or PCT International filing date of the continuation-in-part application, if applicable.

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate, or of any PCT International application having a filing date before that of the application on which priority is claimed.

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I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below.

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Application Number(s)	Filing Date (MM/DD/YYYY)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) or § 365(c) of any PCT international application designating the United States of America, listed below.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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